# PATENT ABSTRACTS OF JAPAN

(11)Publication number :

07-033419

(43)Date of publication of application: 03.02.1995

(51)Int.Cl.

CO1B 31/02

B01D 46/02 C01G 1/00

(21)Application number: 05-199083

(71)Applicant: RITSUMEIKAN

(22)Date of filing: 16.07.1993 (72)Inventor: KAKIUCHI CHIHIRO

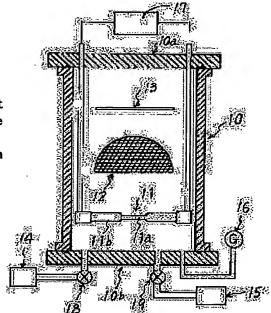
NAKAYAMA YASUYUKI

# (54) METHOD AND APPARATUS FOR COLLECTING CARBON 60

# (57)Abstract:

PURPOSE: To easily and efficiently capture C60 molecules by arranging a filter which allows passage of the C60 molecules but captures amorphous carbon particles and a collecting means for sticking the crystals of C60 in a specific manner.

CONSTITUTION: An inert gaseous atmosphere of about 100Torr is formed within a bell-jar 10. Smoke contg. the C60 molecules of a high concn. past a first net 12 rises together with the convection of the gas and arrives at a second net 13. The temp. of the smoke is about 300 to 350° C in the region of the second net 13 and is the temp. suitable for crystallization of the C60 molecules. Then, the C60 molecules are crystallized on the second net 13 and are stuck to the net 13. The net 13 is recovered at the point of the time when the specified amt. of soot sticks to the second net 13. The soot of the net 13 is dropped by a brush and is collected, by which the C60 is easily collected. The amorphous carbon particles and the C60 are easily separately



collected according to the height from carbon electrodes according to such device.

### \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

#### **CLAIMS**

#### [Claim(s)]

[Claim 1] Into an inert gas ambient atmosphere, arrange the carbon electrode for arc discharge of a pair, and arc discharge is performed. To the temperature field which the molecule of the carbon 60 in the convection current of the smoke generated from the carbon electrode of said pair does not crystallize The molecule of said carbon 60 arranges a filter means to catch an amorphous carbon particle although you make it pass. The approach for collecting the carbon 60 characterized by having arranged a collection means to make the crystal of said carbon 60 adhere to the temperature field which is the downstream of said filter means in the convection current of said smoke, and the molecule of carbon 60 crystalizes.

[Claim 2] The approach according to claim 1 characterized by having arranged said filter means to the field with a temperature [ in the convection current of said smoke ] of 400-500 degrees C, and having arranged said collection means to the field with a temperature [ in the convection current of said smoke ] of 300-350 degrees C.

[Claim 3] Said filter means is an approach according to claim 1 or 2 characterized by consisting of a network.

[Claim 4] A vacuum tub and the carbon electrode for arc discharge of the pair arranged inside said vacuum tub, A filter means to catch an amorphous carbon particle although the molecule of carbon 60 arranged above said carbon electrode in the external power connected to the carbon electrode of said pair and the interior of said vacuum tub is made to pass, The collection means to which the crystal of the carbon 60 arranged above said filter means in the interior of said vacuum tub is made to adhere, It has the vacuum pump connected to said vacuum tub, the inert gas supply means connected to said vacuum tub, and the vacuum gage connected to said vacuum tub. Said filter means It is arranged to the temperature field which the molecule of the carbon 60 in the convection current of the smoke produced from the carbon electrode of said pair at the time of arc discharge does not crystallize. Said collection means Equipment for collecting the carbon 60 characterized by being arranged to the temperature field which the molecule of the carbon 60 in the convection current of said smoke crystalizes.

[Claim 4] Said filter means is equipment according to claim 3 characterized by consisting of a network.

[Claim 5] Said network is equipment according to claim 4 characterized by arranging the shape of a dome so that opening of nothing and said dome may overlook said carbon electrode.

[Claim 6] Said filter means is equipment according to claim 3 to 5 characterized by being arranged to a field with a temperature [ in the convection current of said smoke ] of 400-500 degrees C, and arranging said collection means to the field with a temperature [ in the convection current of said smoke ] of 300-350 degrees C.

JPO and NCIP! are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

[Description of the Prior Art] carbon 60 (henceforth "C60") -- K3 C60 (19.28) and K2

- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

#### **DETAILED DESCRIPTION**

# [Detailed Description of the Invention] [0001]

RbC60 (21.8) (the inside of a parenthesis is transition temperature (K)) etc. -- transition temperature Tc In order to form a hot superconductivity salt relatively, it is observed in recent years and the approach for collecting C60 is already learned. According to this method of collecting C60, soot is first collected by gas evaporation at first. In soot, C60 molecule is contained about 10%, and about 90% of remainder is an amorphous carbon particle. The collected soot is melted in organic solvents, such as benzene and toluene. Although an amorphous carbon particle does not melt into an organic solvent at this time, C60 molecule melts into an organic solvent. Therefore, it is refined after C60 molecule is separated by removing the organic solvent into which C60 molecule melted. [0002] The collection of the soot by gas evaporation is simply explained to below. The carbon rod for arc discharge of a pair is arranged in a bell jar, arc discharge is performed in the ambient atmosphere of the inert gas of about 100 Torr(s), and a carbon rod is heated. At this time, a carbon rod reaches the temperature of 3000 degrees C or more, the carbon steam generated from the carbon rod near the carbon rod collides [ the gas molecule of an ambient atmosphere, and ], condenses it, and it makes a cluster, and a steam adsorbs and carries out vapor growth of it on a cluster, and it serves as a solid particulate (soot). And soot goes up with the convection current of gas, and forms smoke. This soot is made to adhere to the glass plate arranged to a suitable means, for example, the medial surface of a bell jar, or the upper part of a carbon rod, and is collected.

[0003] However, effectiveness in this collecting method's requiring time and effort very much is very bad, and the present condition is that it is utmost to collect about 100-150mg of C60 molecules by the activity on the 1st. For this reason, in current and a commercial scene, 1g carries out C60 molecule of tens of thousands of yen.

[0004]

[Problem(s) to be Solved by the Invention] Therefore, the technical problem of this invention is enabling it to offer the approach and equipment which can collect C60 molecule simply and efficiently that the fault of the conventional method of collecting C60 should be canceled, and to offer C60 molecule cheaply by it.
[0005]

[Means for Solving the Problem] this invention person measured the temperature of the smoke generated from a carbon rod as a function of the height from a carbon rod in gas evaporation. The temperature of smoke discovered falling rapidly as smoke goes up, and falling even to extent of the temperature which C60 molecule already crystallizes from a carbon rod at a point with a height of about 60mm as a result of measurement. And with regards to the height from a carbon rod, it hit on an idea of C60 and an amorphous carbon particle by using this temperature characteristic for a separation collection to be carried out easily.

[0006] Namely, into an inert gas ambient atmosphere, this invention arranges the carbon

electrode for arc discharge of a pair, and performs arc discharge. To the temperature field which the molecule of the carbon 60 in the convection current of the smoke generated from the carbon electrode of said pair does not crystallize The molecule of said carbon 60 arranges a filter means to catch an amorphous carbon particle although you make it pass. The approach for collecting the carbon 60 characterized by having arranged a collection means to make the crystal of said carbon 60 adhere to the temperature field which is the downstream of said filter means in the convection current of said smoke, and the molecule of carbon 60 crystalizes is constituted.

[0007] At this time, it is desirable to arrange said filter means to a field with a temperature [in the convection current of said smoke] of 400-500 degrees C, and to arrange said collection means to a field with a temperature [in the convection current of said smoke] of 300-350 degrees C. Moreover, in order to attain a filter function, without disturbing the convection current of gas, i.e., the convection current of smoke, as much as possible, as for said filter means, consisting of a network is desirable.

[0008] Furthermore, the carbon electrode for arc discharge of the pair arranged inside a vacuum tub and said vacuum tub as equipment suitable for enforcing the above-mentioned approach, A filter means to catch an amorphous carbon particle although the molecule of carbon 60 arranged above said carbon electrode in the external power connected to the carbon electrode of said pair and the interior of said vacuum tub is made to pass, The collection means to which the crystal of the carbon 60 arranged above said filter means in the interior of said vacuum tub is made to adhere, It has the vacuum pump connected to said vacuum tub, the inert gas supply means connected to said vacuum tub, and the vacuum gage connected to said vacuum tub. Said filter means It is arranged to the temperature field which the molecule of the carbon 60 in the convection current of the smoke produced from the carbon electrode of said pair at the time of arc discharge does not crystallize. Said collection means The equipment for collecting the carbon 60 characterized by being arranged to the temperature field which the molecule of the carbon 60 in the convection current of said smoke crystalizes is constituted.

[0009] Preferably, said filter means consists of a network. Moreover, as for said network, it is desirable to arrange the shape of a dome so that opening of nothing and said dome may overlook said carbon electrode. Furthermore, said filter means is arranged to a field with a temperature [ in the convection current of said smoke ] of 400-500 degrees C, and it is [ said collection means ] desirable to be arranged to the field with a temperature [ in the convection current of said smoke ] of 300-350 degrees C.

[0010]

[Function] In the configuration of this invention, the smoke generated from the carbonaceous Plastic solid goes up with the convection current of gas. And although most amorphous carbon particles contained in smoke are caught by the filter means when smoke passes a filter means, C60 with a molecule condition passes a filter means, without crystallizing. The smoke which contained C60 so much goes up further with the convection current of gas, and reaches a collection means. And it crystallizes and the molecule of C60 adheres to a collection means here.

[0011]

[Example] Hereafter, the desirable example of this invention is explained with reference to an accompanying drawing. Drawing 1 is a schematic diagram explaining the approach for collecting C60 by this invention. In drawing 1, if the carbon electrode 1 of a pair is arranged in the ambient atmosphere of the inert gas of about 100 Torr(s), for example, is heated by arc discharge, a carbon electrode 1 reaches the temperature of 3000 degrees C or more, the carbon steam generated from the carbon electrode 1 near the carbon electrode 1 will collide [ the gas molecule of an ambient atmosphere, and ], will condense it, and it will make a cluster. And a carbon steam adsorbs, vapor growth is carried out, and it becomes a solid particulate (soot) at a cluster top, and this soot goes up with the convection current of the gas by heating, and forms smoke 2.

[0012] If the temperature of smoke 2 is measured as a function of the height from a carbon electrode 1 at this time, it will become like the graph shown in <u>drawing 3</u>. Along with the rise of smoke 2, it turns out that the temperature of smoke 2 falls rapidly so that clearly from this graph. If this temperature characteristic is used, with regards to the height of a carbon electrode 1, the separation collection of an amorphous carbon particle and C60 can be carried out.

[0013] That is, although the temperature field which the molecule of C60 in the convection current of smoke does not crystalize is made to pass the molecule of C60, an amorphous carbon particle arranges a filter means 3 to catch, and arranges a collection means 4 to make the crystal of C60 adhere to the temperature field which is the downstream (upper part) of the filter means in the convection current of smoke further, and the molecule of C60 crystalizes. In this case, the 1st network which has the mesh of magnitude with it is used. [ what can attain an expected filter function, without if possible the filter means 3 disturbing the convection current of smoke desirable for example, and ] [ fixed ] Moreover, although the collection means 4 may be what kind of thing as long as it makes the crystal of C60 adhere, the 2nd network with which what does not disturb the convection current of gas if possible has the mesh of fixed magnitude preferably like the filter means 3 is used. [0014] In this way, the smoke generated from the carbon electrode 1 goes up with the

convection current of gas. And although most amorphous carbon particles in smoke adhere to the 1st network 3 and it is caught when smoke passes the 1st network 3 (filter means), the molecule of C60 passes the mesh of the 1st network 3, and goes up further toward the 2nd network 4. And it crystallizes on the 2nd network 4 and adheres to a network 4. If the 2nd networks 4 are collected and adhering soot is dropped and collected by the brush when the soot of a constant rate adheres to the 2nd network 4, C60 will be obtained simply. In addition, what is necessary is just to drop suitably the soot which adhered by the brush etc., when the 1st network changes into a blinding condition in the middle of a collection.

[0015] In order to check whether the separation collection of an amorphous carbon particle and C60 is actually carried out certainly by the above-mentioned approach, the electron microscope image and diffraction figure of the collection object by the 1st network 3 and the collection object by the 2nd network 4 were investigated. Consequently, most collection objects by the 1st network are amorphous carbon particles, and it turned out that most collection objects by the 2nd network are C60 crystallized.

[0016] One example of the equipment suitable for enforcing the approach by this invention was shown in drawing 2. As shown in drawing 2, equipment has the cylinder-like bell jar 10. The carbon rod 11 for arc discharge of a pair is arranged horizontally, and is supported by the lower part in a bell jar 10 at top-cover 10made from stainless steel a of a bell jar 10. The apical surface which one carbon rod 11a is formed in so that it may become a path with a point smaller than the remaining part, and the carbon rod 11 of a pair counters touches mutually. In this case, the apical surface of the carbon rod 11 of a pair sets suitable spacing, and may be counterposed. The carbon rod 11 of a pair is connected to the external power 17 for discharge.

[0017] Above the carbon rod 11, inside a bell jar 10, the 1st dome-like network 12 made from stainless steel makes a carbon rod 11 face the opening, and is arranged, and it is supported by the suitable (illustration is not carried out) member at top-cover 10a of a bell jar 10. The 1st network 12 has an about 2-3mm mesh. Above the 1st network 12 in the interior of a bell jar 10, the 2nd plate-like network 13 made from stainless steel is arranged horizontally, and is supported by the suitable (illustration is not carried out) member at top-cover 10a of a bell jar 10. The 2nd network 13 has about 1mm mesh.

[0018] The 1st network 12 and 2nd network 13 are arranged in the height location beforehand determined from the temperature characteristic of the smoke produced from a carbon rod 11 at the time of arc discharge, respectively. Namely, as for the 1st network 12, the temperature of smoke is arranged in the height location where the temperature of smoke becomes about 400-500 degrees C at the location used as about 300-350 degrees C, as for

the 2nd network 13.

[0019] The inert gas storage equipment 15 for introducing inert gas into the interior of the vacuum pump 14 for lengthening the interior of a bell jar to a vacuum and a bell jar is connected to lower lid 10b of a bell jar 10 through the bulb 18 and the bulb 19, respectively. Furthermore, the vacuum gage 16 for measuring the degree of vacuum inside a bell jar is connected to lower lid 10b of a bell jar 10.

[0020] Hereafter, the actuation approach of this equipment is explained. First, at first, a vacuum pump 14 is made to operate and the interior of a bell jar 10 is lengthened by the vacuum. When the interior of a bell jar will be in a suitable vacua, while a bulb 18 is closed, a bulb 19 is opened wide and inert gas is introduced into the interior of a bell jar from inert gas storage equipment 15. At this time, the gas pressure of the inert gas introduced is measured with a vacuum gage 16. In this way, the inert gas ambient atmosphere of about 100 Torr(s) is formed in the interior of a bell jar 10.

[0021] Then, arc discharge is performed. By arc discharge, a carbon rod 11 goes up toward the 1st network 12 with the convection current of the gas which it was heated by 3000 degrees C or more, and smoke occurred from the carbon rod 11, and occurred inside the bell jar. The temperature of smoke is about 400–500 degrees C in the field of a network 12, and C60 is still in the condition of a molecule at this temperature. Therefore, although C60 molecule passes the mesh of the 1st network 12 and goes up further, an amorphous carbon particle adheres to the 1st network 12. In this way, most amorphous carbon particles in smoke are removed by the 1st network 12.

[0022] The smoke containing C60 high-concentration molecule which passed the 1st network 12 goes up with the convection current of gas, and reaches the 2nd network 13. The temperature of smoke is about 300-350 degrees C in the field of the 2nd network 13, and is the temperature suitable for crystallization of C60 molecule. Therefore, it crystallizes on the 2nd network 13 and C60 molecule adheres to a network 13. When the soot of a constant rate adheres to the 2nd network 13, networks 13 are collected, and C60 is simply obtained by dropping the soot of a network 13 on a brush and collecting it.

[0023] Thus, according to this invention, with regards to the height from a carbon electrode, the separation collection of an amorphous carbon particle and C60 can be carried out easily. Consequently, an activity [ say / separating an amorphous carbon particle and C60 from soot using an organic solvent ] like before which is inefficient—like and wastes time amount becomes unnecessary. And it becomes possible to collect C60 efficiently very simply. [0024] In addition, in this example, although C60 was collected in the condition that there are no receipts and payments of gas to a bell jar, as the interior of a bell jar is set to about 100 Torr(s), C60 can also be collected for gas with a sink, making the pumping rate and the amount of gas installation which lead gas from a top cover and lead gas balance, while introducing gas from inert gas storage equipment. In this case, at once more many C60 is collectable.

[0025]

[Effect of the Invention] Since the separation collection of C60 and the amorphous carbon particle can be carried out based on the temperature characteristic of the smoke which heated the carbonaceous solid-state in the inert gas ambient atmosphere, and was produced by it according to this invention, it becomes unnecessary as mentioned above, to do the separation activity of C60 from soot using an organic solvent like before. And it becomes possible to collect C60 very simply and efficiently, and, therefore, C60 can be cheaply offered now.

#### \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

#### **DESCRIPTION OF DRAWINGS**

[Brief Description of the Drawings]

[Drawing 1] It is drawing which explained roughly the approach for collecting C60 by this invention.

[Drawing 2] It is drawing of longitudinal section having shown roughly one example of the equipment for collecting C60 by this invention.

[Drawing 3] In the situation shown in <u>drawing 1</u>, it is the graph which plotted the temperature of the smoke produced from a carbon electrode as a function of the height from a carbon electrode.

[Description of Notations]

- 1 Carbon Electrode for Arc Discharge
- 2 Smoke
- 3 1st Network (Filter Means)
- 4 2nd Network (Collection Means)

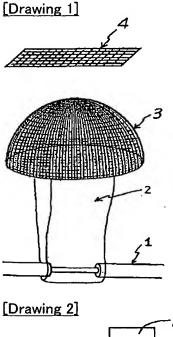
[Translation done.]

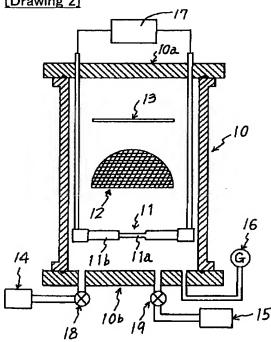
# \* NOTICES \*

JPO and NCIPI are not responsible for any damages caused by the use of this translation.

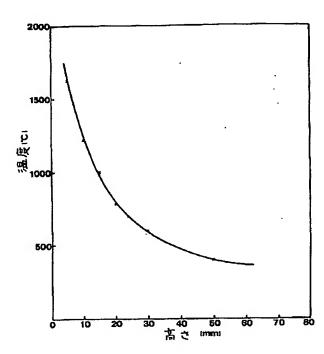
- 1. This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

# **DRAWINGS**





[Drawing 3]



[Translation done.]